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Textile Waste Liquor Disposal

THE studies on the Disposal and Recovery of Textile Waste Liquors that are being financed by the Textile Foundation, and that have been conducted for some two years under the direction of Dr. H. G. Baity, dean of the School of Engineering, University of North Carolina, are now being expanded to cover the waste liquors of all textile mills. Preliminary to further study a survey of textile waste liquor treatment will be conducted under the direction of John C. Geyer with William A. Perry as research assistant. Mr. Geyer is planning to visit as many mills as possible throughout the country that have liquid waste disposal or recovery installations, and representatives of mills who are interested in this problem. His survey will be materially aided and made more resultful if those manufacturers who are interested in the subject will address him at Drawer 432, Chapel Hill, N. C.

A large amount of laboratory and field work has been conducted on the disposal and recovery of liquid wastes of dye houses and print works in North Carolina by Maxwelton S. Campbell, working under the direction of Dr. Baity. Mr. Campbell's final work was conducted in an experimental waste disposal plant, financed by the Textile Foundation, operating upon color shop wastes from the Proximity Print Works, Greensboro, N. C. The four reports of Mr. Campbell's work, as published in *Textile Research*, have been reprinted and copies may be obtained by addressing the editor, or Secretary, Textile Foundation, Commerce Bldg., Washington, D. C.

Completed work has been resultful in determining the most economical methods of treating liquid color shop wastes of cotton mills to dispose of them without damaging the streams or sewage treatment plants into which they may be discharged. Except in a few instances, however, study of economical methods of recovering liquid wastes has not been very resultful. Nevertheless, it is realized that recovery methods and reduction of processing wastes are important features of these studies.

The complete survey of liquid waste treatment that is now to be conducted in all branches of the textile industry is regarded by the board of directors of the Textile Foundation as essential to the planning of future work, and to prevent additional work from duplicating or overlapping work that has been completed elsewhere in this country or abroad. Following the completion of the survey a report will be published summarizing current knowledge of the means of disposal of liquid wastes throughout the textile industry. This report will also include information concerning the laws and boards governing stream pollution, which exist or might be set up, as well as existing general and scientific information. The report will be written in language equally understandable by the manufacturer, engineer or research worker.

Micro-Analysis of Textile Fibres

Part IV.—Observations on the Structure of Flax, Manila and Jute

By G. GORDON OSBORNE *

(*Continued from Vol. V, No. 8*)

It would be extremely interesting and very worthwhile to show by means of photomicrographs the fissure openings in a single Manila cell. This has not been easy since the breaks that occur and can be seen are rare, as they must have openings parallel to the line of sight. Furthermore, the magnification must be high and, even though it is possible to view the specimen clearly, it is hard to obtain the same results photographically. Such a specimen is, however, illustrated in Fig. 17. The pictures were taken with a 1.8 mm. oil immersion objective and a 10x ocular from a Canada balsam mount between crossed nicols and with a first order red selenite.

Fissure Openings

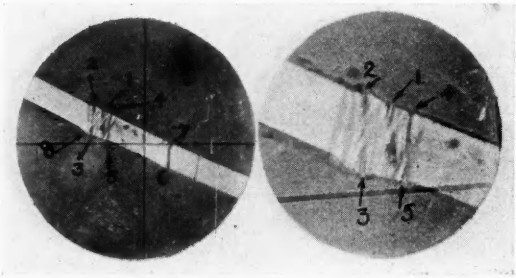
Fissure openings can be seen between crossed nicols with or without a selenite. A first order red plate, however, seems to help the detail in the image. A close inspection of the portion of the cell showing these openings or breaks in the edge of the wall will reveal that they are decidedly appreciable. They can be seen clearly during rotation of the cell about the axis of the microscope until the increasing brightness of the cell itself renders them so obscure as to be indeterminate. These fissure-cracks or openings extend further into the cell than one view would indicate, as can be shown by casts or changes in focus (see Fig. 22B) and in some instances they run almost all the way across to the opposite side.

Two different magnifications are shown for the same specimen in Fig. 17. Except for this, conditions for each were identical. At A a clear opening is shown at points 5 and 2 with blurred openings at 1 and 4. Opening 2 is much more prominent in the enlargement B, where the actual crack as such is visible for a short distance and the continuation of the fissure is indicated by the dark, blurred lines running inward from it. A considerable over-hang or off-set is noticeable to the right of this break. Point 5 in enlargement B is not quite so clear, but it can be seen that its opening is even wider than that of 2.

In A, fissure openings at all the indicated points are quite clear, and in the enlargement B each opening is so large as to be measurable. Thus, from this view the largest opening (1) is 0.8 μ, and the smallest at 4 is 0.3 μ. Of course, this may not be either the maximum or minimum distance, but

* Mr. Osborne, as a Senior Fellow of the Textile Foundation, worked under the direction of Prof. E. R. Schwarz, Massachusetts Institute of Technology, Cambridge, Mass.

it is substantially correct for this position of focus. There is the possibility that 4 and 5 are really but one fissure which extends completely across the cell. Setting the focus on the top surface of the cell appears to confirm this contention. The bulge 6, between the angling fissures, the



A B

FIG. 17. A.—Surface fissures in flax cell. B.—Left portion of A at higher power. (Elliptically polarized light.)

overhang, or jut 7, at the fissure opening, and the enlargement 8 are all evident.

Flax Cell Casts

After dissecting a fibre bundle, the largest, most perfect cells were selected for casting. The technique used follows in all respects that reported previously by the author * with the single exception that the casting medium was changed, celluloid in acetone being substituted for collodion.

Fig. 18A is a camera-lucida sketch of a single flax cell mounted in celluloid. Two small joints are included in this mount, which is relatively free from imperfections elsewhere. Only the coarsest and most prominent fissures in the joints are recorded in the sketch, while the cell length between them contains only a few fine markings.

The cast of this cell shows decided evidence of surface openings at both joints. Correlation between size and shape, as previously explained,* is not perfect, but the relative position of these imperfections in cell and cast is excellent. There is little evidence of surface fissuring in the body of the cell between the joints, and this is slightly different than that indicated in the cell itself.

Fig. 18B shows another cell and its cast. The cell is marked by numerous fissures, although these are of a fine nature. The heavy group at each end of the cell has left a definite impression in the cast and correlation between the two is good. The small fissures throughout the cell also have a good correlation with their cast impressions, and, as is to be expected, all impressions recorded in the cast are shorter and finer than the originals.

* *Tex. Rsch.*, *V*, 2, 84 (1934).

Fig. 18C offers the best correlation between cell and cast of the three. This is true not only as to position, but also for shape and size.

Fig. 19 shows a somewhat similar comparison. Here, due to a stroke of luck, an extra check on the precision of the work is offered. Instead of selecting a single cell for mounting, as was intended, two flax cells were inadvertently taken. As soon as they were inspected at high power this was apparent, but the experiment was continued nevertheless. On removal from the medium only the larger of the two cells was taken out, the other remaining in the cast. Complete assurance of this was obtained

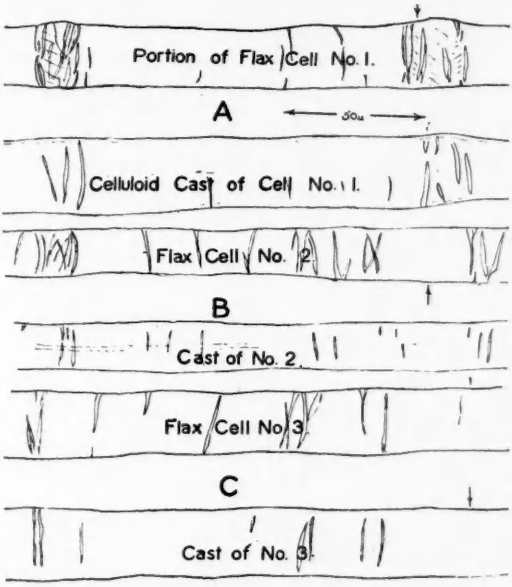


FIG. 18. Three flax cells with the celluloid cast accompanying each.

by rotating the specimen between crossed nicols. This shows by changes in color and lighting whether any fragments of fibrous materials remain, the material of the cast being isotropic. The entire length of the smaller cell seemed undisturbed while the large one was completely removed. Therefore, on each drawing of Fig. 19 there are three cells and one cast illustrated. Different portions of the same cells, rather than three different cells as in Fig. 18, are shown. The accuracy of the work is shown by comparing the detail of the two smaller cells in each view.

Fig. 19A shows the two cells lying side by side in the casting medium. Correlation as to size and position is good for the right-hand group of fissures; the other group, however, failed to register. The same arrange-

ment is shown at Fig. 19B. Here the similarity between cast and cell is very good, and there is a much higher percentage of fissure impressions recorded in the cast. Fig. 19C is also good, although the impressions left in the cast are not grouped as in the original cell.

Observations from the work done on flax further emphasize the fact that perfection of correlation is most likely of attainment when the specimen is large and the surface markings wide and deep. Thus, the best results are given in Fig. 18C, where the largest cell was used and the fissures were wide. Conversely, the fissures shown in Fig. 19C have little chance of being filled by, or of retaining, any of the casting medium.

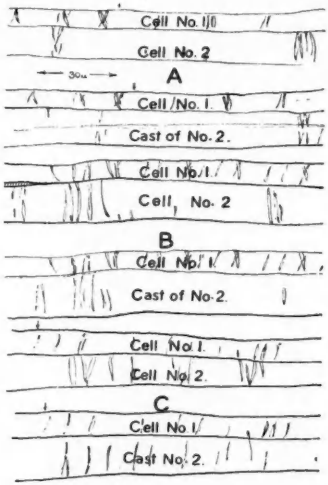


FIG. 19. Flax cells with cast, showing surface fissures.

It is interesting to note that the angle made by the fissure imprints in the casts with the cell axis tends towards a right angle, and is considerably less than the angle obtained by averaging all markings.* This may be explained as being due to the fact that all the impressions recorded are surface breaks left by surface fissures, while the general average was made from all observable types in the cell itself. Also noticeable is the fact that those fissures which are close together are nearly parallel; and, as would be expected, there is no recording of crossings or "x" formations.

The parallel double lines down the center of the cast of Fig. 19A are caused by cell removal, while the arrows are reference points. The casting of fibre bundles, rather than single cells, does not seem to be possible. Few, if any, fissures register in the cast, and these, due to the great mass of extraneous detail, are not easily interpretable.

* The same is true in ramie.

If the flax cell were split longitudinally and a cast made of its inner surface, there is a strong likelihood that the presence of internal fissure would be shown. Such being the case, its construction could be interpreted as similar to that of ramie which is illustrated diagrammatically in a previous article by the author,* and would be subject to the same theory concerning strength.

Manila and jute could be treated in like manner, but to-date the difficulty of handling short length cells by this technique has prevented any casting. However, from their behavior and microscopic appearance there seems little doubt but that these too are subject to similar fissuring.

Figs. 20, 21 and 22 each illustrate highly magnified views of longitudinal sections of the respective single cells of Manila, jute and flax. All

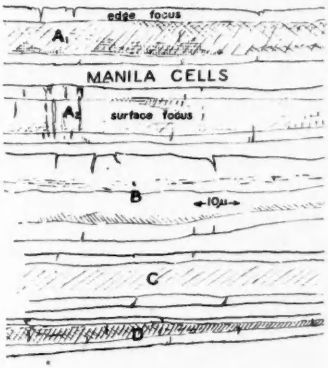


FIG. 20. Manila cells showing lumen, wall thickness and surface fissure formations.

the figures are camera-lucida drawings of balsam mounts between crossed nicols and a first order red selenite plate.

Manila Cells

Fig. 20A (of Manila hemp) shows the same section of a single cell at two different focus points. Drawing No. A1 is taken with the extreme edge of the cell in sharp focus. The small jagged breaks through the cuticle are very clear, but, except for this evidence of trans-fibre fissuring, the rest of the section is devoid of detail. In No. A2 the focus has been slightly raised so that it now centers on the upper curved surface of the cell. The change is at once apparent; a multitude of fissures spring into view, and these are at the exact spot where the most prominent breaks occurred before. The correlation between the two views is excellent. It will be noticed here that the fissures are almost perpendicular to the longitudinal axis of the cell.

* *Tex. Rsch.*, *V*, 2, 89 (1934), Fig. 9.

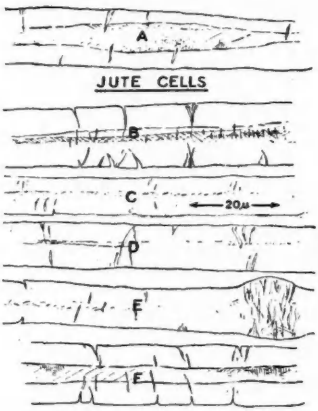


FIG. 21. Jute cells showing typical surface fissures.

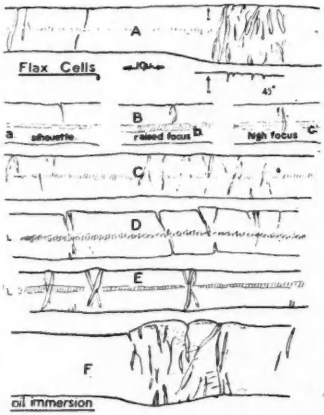


FIG. 22. Flax cells showing fissures and "cross-marking." (See Fig. 23.)

Fig. 20B illustrates the same phenomena. In this case the breaks are very clean and a characteristic off-set, or overhanging edge, is noticed beside each of them. Fig. 20C illustrates a normal, or average, portion of a single cell. Here there are few trans-fibre fissures and these are small. The cell walls are cylindrical, smooth, and devoid of other visible detail. Fig. 20D shows a normal portion close to the tip of the cell.

Jute Cells

The fissures, in general, are small and short and are not numerous. They are scattered at random along the cell. The actual openings in the cell wall are not large. The joints or nodes are also few in number and are not prominent. There is no appreciable swelling or enlargement where they occur, and, except for the increase and concentration in the number of penetrating fissures, the appearance and structure are as in the body of the cell. In these nodes the fissures are almost always perpendicular to the cell axis, while in the rest of the cell they make an angle of approximately 5° with the longitudinal axis, but the variations from this are large.

The normal appearance of the cell is as in Fig. 21C, with an occasional node or group of fissures as in Figs. 21A and 21B. The sketches illustrate the regularity in size of the wall and lumen, although, due to the shade and shadow effect, the lumen diameter is probably exaggerated at the expense of wall thickness. The cell's straightness, smoothness and freedom from defect and irregularities are also well brought out. At powers lower than oil immersion, and with ordinary transmitted light, the single cell appears light grey with the trans-fibre fissures showing as dark black lines or cracks.

Fig. 21A shows a typical longitudinal view of a single jute cell. In the central portion a small fissure-break into the cell wall is visible and the presence of further random fissuring along the cell is recorded. Fig. 21B shows a densely-fissured portion of a cell. Here the fissure-breaks are large and particularly deep, extending in well towards the lumen, which is decidedly narrow. There is a slight off-set, or overhang, by each fissure on the upper side, which breaks the smooth, straight line of the cell wall. Those in the under side are shorter but wider with typical inclination and crossings. Fig. 21C is a position close to the tip of the cell; one fissure-break with decided jutting-out of the cell wall and numerous randomly arranged small fissures are evident. Fig. 21D is another typical longitudinal view, heavily fissured and with narrow lumen. Fig. 21E illustrates a joint, or knot, which appears to be literally riddled with fissures and other imperfections. These are in such dense order that it is impossible to sketch any one of them with precision. The decided enlargement, or swelling, is evident, and at this place it is impossible to produce more than a slight degree of extinction between crossed nicols. Furthermore, all trace of the lumen is lost and it is not possible to tell whether it enlarges, contracts, or stops altogether at these points. Actual fissure-breaks can be seen in this region by carefully focussing on the edge, but they are small in size and number. Two large imperfections of this kind, however, are shown at the other end of the figure, where the wall off-set, or break, is so great as to resemble the scale projection characteristic of wool fibre. Fig. 21F is an excellent view, in sharp focus, of a frequently-fissured cell wall. The fissure openings, arrangement and angularity are typical.

Fig. 21, as a whole, shows the smoothness and regularity of the outside wall, and the variation in the size of the lumen. The cells themselves are fairly heavily fissured; considerably more so than Manila and slightly more so than in flax. This must be partially discounted, however, as the sample from which these fibres were taken was further advanced in mechanical treatment than the others. The fissures themselves, although numerous, are fairly fine, and do not extend over a great distance. There is a decided

degree of angularity between them and the fibre axis. This averages approximately 6°, but the variations from it are wide.

The typical longitudinal, single-cell view is probably best represented by Fig. 21A, while the densely fissured portions are best shown in Fig. 21F. In ordinary transmitted light the cell appears bright; the trans-fibre fissures appear clearly as fine black lines.

Flax Cells

Fig. 22A, showing longitudinal views of flax cells, is taken between crossed nicols with an oil immersion lens, as for the Manila and jute, the fibre cell being parallel to the plane of the nicol, and shows a normal portion of a single cell and a joint. This joint appears as an enlargement in the cell wall and is replete with structural detail. Most of this seems to be composed of external and internal fissures of all shapes and sizes. The lumen here is obscured and the detail cannot be precisely shown. As indicated, there is very little detail seen along the edge. If, however, the cell is rotated to a position at 45° with the planes of polarization, the detail along the edge becomes much clearer. This is illustrated directly below Fig. 22A, and the actual penetration of the fissures into the cell wall is apparent. The reference point "x" serves for purposes of direct comparison.

Fig. 22B shows the great variation that can be produced by focus. At "a" is illustrated a cell wall in sharp "silhouette" side focus. The penetrating fissure is extremely clear, with good detail of the wall overhang and opening. In "b" the focus has been slightly raised, producing a great change in the appearance of the specimen. The opening in the wall is no longer apparent. The fissure has increased in length and breadth, and an additional smaller imperfection is noticed. In "c" the change is again as great. Length and breadth have again changed, while the small fissure becomes more prominent. Contact with, and the opening in, the outer wall are not evident due to loss of focus. These changes, of course, are due to the cylindrical shape of the cell, and to the change in focus necessitated as different portions of its curved surface are viewed.

In somewhat the same manner of careful focusing, it can be shown that the majority of fissures which seem to be angularly opposite are, in fact, the same, and this appearance is also due to the cylindrical shape of the cell. Thus, those fissures which slope upward to the right in Fig. 22C are on the surface of the cell, while those that point downward to the left are on the under side of the specimen. Their apparently contradictory appearance is due to the extreme thinness of the cell itself between the bottoms of these fissures. This is so fine that when the focus is directed on the top of the specimen the under side also is visible and vice versa.

Fig. 22C is a fairly typical portion of a fissured cell wall. Fig. 22D is more drastically fissured. Here the fissures are wide and heavy. The cell wall off-sets are most prominent, so much so as to give the cell an almost joint-like structure.* Fig. 22E shows a section of a cell with the characteristic "cross" markings so often mentioned in the literature.[8, 9] Fig. 22F shows an extra large joint or knot. It is impossible to record all the detail, so much of it has been sacrified to secure the small portion that

* The similarity between this outline, and the view of the old Egyptian flax fibre in polarized light given by Herzog,[7] is very striking.

is drawn. Again, the predominant part played by the fissures in this structure is evident.

The normal cell is best represented by Fig. 22C. Its outline is not as smooth or regular as either Manila or jute. The fissures are long and thin, making an angle of approximately 9° with the fibre axis. In amount, the fissuring is slightly less than in jute, although the average fissure is larger. The joint, or knot structure, is more common in flax than in the other two fibres, and the fissuring in these protrusions is denser and more apparent. When the "x" markings appear (and in reality they are heavy fissures) there is a noticeable bulge or swelling between the ends of the two lines forming the crossing of the "x." This is well shown in Fig. 22E. Sometimes these places have the appearance of miniature joints.

It may be helpful to show by diagrammatic sketches the relative position of these cell-wall fissures and to take as the example the "x"-formation. If the cell is conceived as in sketch "A" (Fig. 23), and is then split up

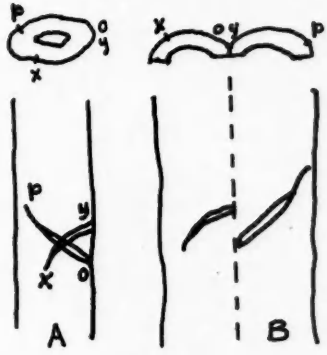


FIG. 23.

its vertical diameter and spread open, so that each semi-section lies in the same plane, the appearance from the top is as illustrated in "B."

A short statement is needed about the joint, node, or knot formations. The three words do not really describe the structure, but are used for want of better. As has been stated, the detail and construction in these portions are so dense and so complicated as to effectively prevent their study and recording. Moreover, such formations are quite numerous. In Manila these joints occur only rarely and are less complicated than those in jute or flax. There is little or no swelling; the concentrated fissures and those of larger size appear to be mostly in the surface, and at right-angles to the cell axis. There is an almost total absence of the fine, much distorted inner structure that hides so much detail in jute and flax. If it were not for the concentration in size and number of fissures, slight thickening, and obscuring of the lumen, the joint portion would hardly be recognized in the cell length.

In jute, such portions are much more pronounced and occur more often, while in flax they are quite frequent. Their presence is manifest by

a considerable enlargement in the cell wall and a most complicated structure. Again, the effects seem to be greater in flax. It is possible to get only partial extinction between crossed nicols, although the cells themselves give almost perfect parallel extinction. The fissures, both internal and external, are mostly small, but lie at all angles, and the inner portions of the joint appear to be composed of much stressed and distorted material, in no definite form or arrangement.

TABLE 1

SINGLE JUTE CELL

Diameter Outlines

1	2 Typical μ	
Average μ	Total	Lumen
3.50	3.25	.50
4.75	4.00	3.00
6.25	5.50	1.00
7.25	6.25	3.25
8.50	7.00	2.25
9.25	9.25	3.00
10.00	10.00	4.25
10.75	10.75	3.75
12.50	12.50	2.75
13.25	14.25	6.25
14.00	17.00	8.25
15.00	17.25	6.50
15.50	15.25	5.25
16.25	14.25	2.75
15.50	14.00	1.25
14.25	13.25	2.00
12.75	12.25	2.25
11.00	11.00	4.00
9.75	9.75	2.75
9.00	8.00	2.00
8.00	7.00	1.00
6.25	5.25	.75
4.00	3.50	.25
2.00		
Total.... 239.25	230.50	69.00.... Total
Ave..... 10.0	10.0	3.0 .... Ave.

1. Average of ten single cell diameters.
2. Typical single cell, lumen and total diameter in microns.

Diameter Outlines

The data for the diameter outline plots of Figs. 24 and 25 were obtained in the following manner: After dissection, perfect single cells were stained and placed on the stage of a polarizing microscope equipped with a filar micrometer of the movable cross-hair type. By rotating the specimen between crossed nicols it was possible to find the extreme tip of the cell, and, from this point to the opposite end, readings of the cell width and lumen width were taken at regular intervals. These readings were tabulated

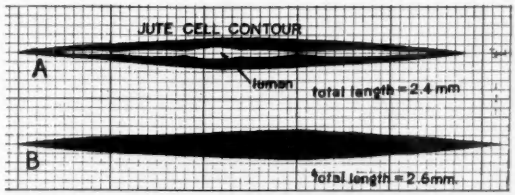


FIG. 24. A.—Typical jute cell contours. Note the irregular lumen diameter. B.—Average cell diameter variation (data from 10 cells).

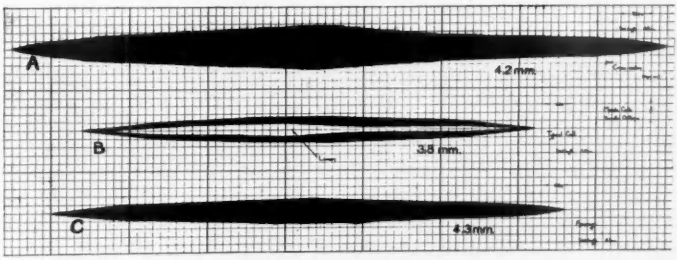


FIG. 25. A.—Manila cell contour determined by measurement of the successive major axes of a set of serial cross-sections. B.—Typical Manila cell showing uniform variation in lumen diameter. C.—Average Manila cell (data from 10 cells).

to correspond to the length along the cell from which they were obtained, and at the finish the total length of the cell was measured to check against the product of the number of readings times the length of interval.

The specimens were mounted in Canada balsam, and at the magnification used (450 ×) the readings secured were fairly fine, four filar micrometer dial graduations equalling 1 micron. There was little difficulty in reading the outside width of the cell, but that of the lumen was not so easy; in cases where any question as to its precision arose it was omitted from the calculations.

TABLE 2

SINGLE MANILA CELL

Diameter Outlines

1	2		3
Average μ	Typical μ		Cross-section μ
	Total	Lumen	
1.75	2.75		4.00
3.75	4.25	1.00	4.75
6.25	6.25	2.00	4.50
9.50	9.50	4.75	5.00
9.25	10.00	6.75	4.00
10.00	9.50	4.00	6.00
10.50	8.50	4.25	8.00
12.00	9.50	6.25	8.50
11.75	13.75	9.75	10.50
12.50	15.00	6.50	13.50
12.25	13.25	8.50	15.50
13.50	15.75	7.00	17.00
13.75	13.75	9.75	16.00
13.50	15.50	8.00	16.75
14.75	15.75	7.75	18.50
15.50	14.75	4.50	18.50
14.25	15.50	3.75	19.00
14.75	11.00	2.00	20.50
13.25	11.25	3.25	22.50
12.25	12.00	2.75	22.50
11.25	10.50	3.50	22.00
11.00	11.25	3.75	25.00
11.25	10.50	3.00	20.00
10.25	9.25	3.75	23.00
9.50	8.50	3.50	22.00
9.75	6.25	3.00	17.00
9.75	2.50	2.75	19.00
8.50	1.50		17.00
8.00			15.50
7.00			15.50
3.00			15.00
2.00			14.00
			12.00
Total..326.25	287.75	124.75	12.50
Ave... 10.2	10.3	4.46	12.00
			13.50
			13.00
			12.00
			10.50
			10.00
			10.00
			6.50
			4.50
			649.50..Total
			14.4 ..Ave.

1. Average of ten single cell diameters.
2. Typical single cell, lumen and total diameter in microns.
3. Diameters taken from serial sections—major axis.

Table 3

Single Manila Cell

Determination of Cross Sectional Areas

1	2	3	4	5	6
Microns	Weight	Weight	Weight	Percent	Area
100	1.2			1. est	.032
150	1.2			3. est	.032
200	2.0	1.9	.1	5.0	.050
250	2.0	1.8	.2	10.0	.050
300	2.3	2.1	.2	8.7	.058
350	2.9	2.7	.2	6.9	.073
400	3.0	2.8	.2	6.7	.076
450	3.2	2.9	.3	9.4	.081
500	4.1	3.8	.3	7.3	.103
600	6.4	5.6	.8	12.5	.161
700	7.5	6.8	.7	9.3	.189
800	8.2	6.1	2.1	25.6	.206
900	8.9	7.0	1.9	21.3	.224
1000	9.3	7.9	1.4	15.1	.234
1100	9.5	7.0	2.5	26.2	.259
1200	9.7	7.7	2.0	20.6	.244
1300	9.7	7.5	2.2	22.7	.244
1400	9.8	7.8	2.0	20.4	.247
1500	11.9	9.6	2.3	19.3	.300
1600	10.8	8.5	2.3	21.2	.272
1700	11.9	9.1	2.8	23.5	.300
1900	13.3	10.4	2.9	21.8	.335
2000	11.9	9.4	2.5	21.0	.300
2100	12.4	9.8	2.6	21.0	.312
2200	13.0	10.2	2.8	21.5	.327
2300	13.2	10.4	2.8	22.2	.332
2500	11.4	9.2	2.2	19.3	.287
2600	11.6	9.5	2.1	18.2	.292
2700	11.4	9.1	2.3	20.1	.287
2800	9.9	8.1	1.8	18.2	.249
2900	8.6	7.4	1.2	13.9	.216
3000	8.0	7.0	1.0	12.5	.201
3100	7.9	7.0	.9	11.4	.198
3200	7.6	7.2	.4	5.3	.192
3300	7.2	5.9	1.3	18.0	.182
3400	6.4	5.4	1.0	15.6	.162
3500	4.0	3.5	.5	12.5	.101
3550	3.7	3.3	.4	10.8	.093
3600	1.9	1.8	.1	5.3	.048
3700	1.1			2. est	.028
3750	.8			3. est	.020
3775	.6			0.	.015
3800	.3			0.	.007
				589.3	7.592 Total
				13.7	.180 Ave.

1. Length along cell in microns.
2. Weight in milligrams of total section.
3. Weight in milligrams of cell wall.
4. Weight in milligrams of lumen.
   (2 − 3 = 4)
5. Percent of area occupied by lumen to total section area.
6. Area in square inches of total camera-lucida sections
   (7 ÷ 2 = 6)
7. 9 sq. in. paper weight 355.6 mg.

TABLE 4

SINGLE JUTE CELL

Determination of Cross Sectional Areas

1	2	3	4	5	6
Microns	Weight	Weight	Weight	Percent	Area
0	.1				.005
50	.2			2.2 est.	.010
100	.5			3.3 est.	.026
150	.5			3.3 est.	.026
200	.8			2.2 est.	.042
250	1.0	.9	.1	10.0	.052
300	1.4	1.3	.1	7.1	.073
350	1.9	1.6	.3	15.8	.099
400	1.9	1.8	.1	5.3	.099
450	2.7	2.4	.3	11.1	.141
500	2.7	2.4	.3	11.1	.141
600	3.9	3.5	.4	10.2	.204
800	4.4	4.0	.4	9.1	.230
900	5.2	4.5	.7	13.5	.272
1000	4.9	4.0	.9	18.3	.256
1100	5.0	4.6	.2	4.0	.262
1200	5.6	5.3	.3	5.4	.293
1300	5.3	5.2	.1	1.9	.279
1400	4.3	3.5	.8	18.6	.225
1500	3.6	3.3	.3	8.3	.188
1600	3.7	3.6	.1	2.7	.194
1700	2.7	2.1	.6	22.1	.141
1800	2.2	2.1	.1	4.5	.115
1900	1.9	1.7	.2	10.5	.099
2000	1.2	1.1	.1	8.3	.063
2100	1.1	1.0	.1	9.1	.058
2150	.9			5.0 est.	.047
2200	.6			2.0 est.	.031
2250	.5			2.0 est.	.026
2300	.2				.010
				Total...225.90	3.707
				Ave.... 7.53	.124

1. Length along cell in microns.
2. Weight in milligrams of total section.
3. Weight in milligrams of cell wall only.
4. Weight in milligrams of lumen (2 − 3 = 4).
5. Percent of area occupied by lumen to total section area.
6. Area in square inches of total camera-lucida sections (7 ÷ 2 = 6).
7. 1 sq. in. of paper weight 19.1 mg.

The data thus secured do not, of course, measure the actual diameter, either maximum or minimum, but merely the width of the cell exposed to direct perpendicular view. Thus the two outlines of Manila hemp taken in this manner are decidedly smaller than the one prepared from measuring the major axis of a complete set of continuous, single-cell cross-sections.

The outline and lumen for the typical case in both illustrations were selected as the most representative of many. Both outside width and lumen are, of course, from the same cell. It will be noticed that, although the cells have the same general overall formation, the lumens differ widely. This has already been noted in reference to the cross-sections, but is even more forcibly evident here. More or less smooth curves have been drawn through the points on the plots; the idea being to show the general, rather than the specific, shape. However, even so, the great variation of the jute lumen, and the much more regular Manila lumen, is apparent. This aspect, taken in conjunction with the cross-sectional illustrations, proves that a consideration of the jute lumen, in the form of a spirally twisted ribbon extending throughout the cell length, as was first speculated, is erroneous. Its final conception must be as an irregularly strangulated, uneven cylinder.

The two average outline graphs are compiled by finding the mean of similar readings from 10 cells of approximately equal length. To allow for slight differences, however, each set of readings was divided at their mid-point, and averages taken from each tip to the middle of the cell. It was not thought worth while to secure a set of average figures for the lumen. The individual cell in outline appears smooth, and averaging equalizes out the jagged edges due to errors of observation. With the jute lumen, however, the variations are distinct and decidedly characteristic, and, as they follow no definite cycle, an averaged set of figures would produce an erroneous result.

Although in some cells there was noticed a tendency for the maximum diameter to be reached and held a little to one side or the other of the mid-point of the cell length, this was not considered or allowed for in the calculations. There is, however, a good likelihood that a maximum size is reached at other than the mid-point. If this is so it is probably regular, but whether such an occurrence is on the apex or basal-point side could not be learned from the samples used, as their direction of growth was not known.

The graphs have greatly changed the dimensional proportions of the cells. The length-to-width ratio for an average Manila cell is 400: 1, and for jute 200: 1. In the illustrations the length has been reduced and the width increased so that the ratios are 13: 1 and 20: 1 respectively.

Referring again to the lumen, two graphs (Figs. 15 and 16 *) have been prepared to show the great variation in the size and changes occasioned in the two species of cell. The data are represented as the percentage of area occupied by the lumen to the total cross-section, and are calculated from the two sets of continuous cross-section figures. It will be seen that in Manila the lumen area is greater and much more regular throughout the length of the cell. The jute lumen is extremely variable, ranging from 2 to over 22% in its mid-section, as compared with a change in Manila from 19 to 25%. Each exhibits approximately the same irregularity in the end portions, but the lumen here is noticeably difficult to measure or draw.

* *Tex. Rsch.*, *V*, 8, 368 (1935).

TABLE 5

Manila Single cell	By weight		By planimeter
Ave. cross sectional area (total)	171** sq. μ	(43)*	.190 sq. in.
Ave. cross sectional area cell wall	157.6 sq. μ	(43)	.162 sq. in.
Ave. cross sectional area lumen	23.4 sq. μ	(43)	.028 sq. in.
Ave. percent of total area occupied by lumen	13.7%	(43)	14.5%
Ave. length	4.1 mm.	(25)	
Ave. width (filar micrometer)	10.2 μ	(320)	
Ave. width (from cross section)	14.4 μ	(45)	(major axis)
Ave. width 'x' cell (filar micrometer)	10.3 μ	(28)	
Ave. width lumen of 'x' cell (filar micrometer)	4.5 μ	(28)	
Jute Single cell			
Ave. cross sectional area (total)	118** sq. μ	(30)	
Ave. cross sectional area cell wall	108.9 sq. μ	(30)	
Ave. cross sectional area lumen	8.9 sq. μ	(30)	
Ave. percent of total area occupied by lumen	7.5%	(30)	
Ave. length	2.4 mm.	(25)	
Ave. width (filar micrometer)	10.0 μ	(240)	
Ave. width 'x' cell (filar micrometer)	10.0 μ	(23)	
Ave. width lumen of 'x' cell (filar micrometer)	3.0 μ	(23)	

* Figure in parenthesis indicates number of measurements from which average was secured.

** $\frac{\text{Average area sq. in.} \times \text{sq. } \mu \text{ per sq. in.}}{2}$ = actual average total area of section in sq. μ.

MAGNIFICATION

e.i. $\frac{.180 \times 25400 \times 25400}{825 \times 825} = 171$ sq. μ.

e.i. $\frac{.124 \times 25400 \times 25400}{825 \times 825} = 118$ sq. μ.

Flax Sections

Typical cross-sections of flax, as studied in this work, appear to be of two types: first, the straight-sided, clear-angled, polygonal sections, as usually described in the literature; and, second, much more irregular and rounded sections. Two factors, either alone or jointly, could affect this: first, the technique of cutting, which, due to compression or shock, may alter the shape of the cell outline; and, secondly, changes may be due to previous treatment, specie of plant and conditions of growth, or the portion of the stem from which the fibre bundle was removed. Of the two, the latter seems the more probable.

At low magnifications all sections appear more alike and somewhat polygonal in shape. At higher power they tend to assume a more rounded

shape. The cell walls are uniform and thick. It will be noticed that the outline is smooth both in curve and straight line, and that the gross-section shape is closer in resemblance to Manila hemp than to jute. The cells are not as orderly in arrangement as those of jute and there are more random spaces in the bundle filled with the pectic middle lamella. This lamella between the cell walls is thick and uniform and forms an even thicker encasing layer around the fibre bundle, thus separating the bundle from the soft bast cell with which it is surrounded.

The lumen is again a valuable diagnostic characteristic. It is small in area; in some cases extremely so; very uniform, and nearly always round or elliptical. Changes in its size and shape are most moderate, and from cell to cell a closely proportional relationship between wall area and lumen is maintained. There is, however, a decided change in all dimensional relationships, depending upon the portion of the plant stem from which the sample is taken. As an example, Herzog [7] lists a range in average cross-sectional area from 75 sq. μ to 450 sq. μ along 75 cm. of stalk, while other writers * frequently refer to the increase in the proportional size of the lumen as fibre removal approaches the root end of the plant.

There is too much variation in dimensions to give more than the approximate range of some. The following, taken from Herzog,[7] after figuring in his usual manner are as follows: length, 10 to 38 mm.; width, 14 to 40 μ; area of section, 75 to 450 sq. μ, and percentage of lumen, 1.3 to 21.0. Perfect cells in the sample used averaged approximately 25 mm. in length, and 20 μ in width.

Cell Tips

Current in the literature of the past is the statement that it is possible to differentiate between bast fibres by the appearance of the tips of the individual cells. This contention has lately been corrected.[10, 11]

There appears to be considerable variation in the shape of the cell tips, but this is not classified enough to serve as a basis for diagnostic prediction. What is interesting, however, is to notice other differences evident in these end regions. In all cases the lumen appears to extend well up into the tip of the cell. In jute it tends to be wider than in Manila and flax, and here the end forms are most variable. Jute tips, however, are noticeably free from fissuring. In this connection Fig. 26 is interesting. It illustrates approximately 500 μ from the tip of the cell down its length. It is very heavily fissured, especially as it approaches its maximum diameter,

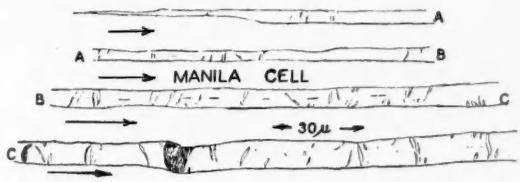


FIG. 26. Manila cell from tip to approximate center.

* See Hanausek,[8] p. 76.

and exhibits one pronounced joint. The lumen extends almost to the end of the very sharp tip and the first group of fissures occurs within 75 μ of this. Generally, even in heavily fissured cells, there is little evidence of this in the 300 μ length back of the tips. Most fissuring is at a maximum in the central portion of the cell and reduces in intensity as it extends outward.

Cell Bundles

Figs. 27, 28 and 29 represent diagrammatically the appearance and arrangement of the cells in the fibre bundle—particularly longitudinally. The drawings were made after a study of the respective single cells, individually, in the fibre bundle, and during dissection; and in conjunction with the previously prepared longitudinal and cross-sectional views.

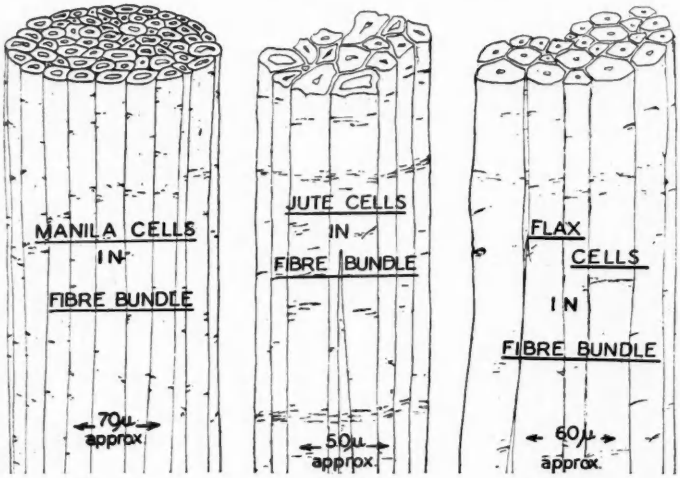


FIG. 27. FIG. 28. FIG. 29.

The Manila hemp bundle (Fig. 27) is the largest and most compact. It is oval or elliptical in shape and contains the greatest number of cells. Under low magnifications the bundle is grey in color with intermittent dark bands running across it. The cells are straight, hard and smooth, and are arranged in the fibre bundle almost as parallel glass rods. Due to the absence of a noticeable middle lamella they appear even closer set and more compact than they actually are.

At higher powers the appearance of the fibre changes but slightly. Very few breaks or imperfections are seen, this possibly due to the great number of cells which tend to obscure detail. The fissures that do appear are at random and are comparatively small. The actual opening is seldom seen and the fissures run but a short way into the cell. There is a tendency for them to form disjointed groupings.

The arrangement of new cells in the bundle is most orderly. In any section they are spaced at maximum distances apart, and only very occasionally do two tips abut or lie close together. This is equally true of the other fibres, but is more noticeable here, due to the greater number of cells and larger area. However, where a cell commences or stops there is a tendency for the regular order of compactness to be disarranged, and the resulting void is filled up with middle lamella.

There appear to be fewer cells per bundle in jute (Fig. 28) than in the other fibres. The shape of the bundle is irregular and variable; the color ranges from almost white to brown; while there is a tendency for the bundles and cells to separate through manipulation. At low powers there is noticed a slight wave in the arrangement of the cells, for they do not lie parallel throughout their length. There are series of marks and fissures on all cells. The majority of these seem to follow one or more definite patterns; either working in helical form around the fibre bundle, or zig-zag backwards and forwards across it at equal intervals. Particularly evident, however, are the lines of fissures which at irregular intervals completely band the fibre bundle. They may be more intense on one cell than another, but because of their prominence they cannot go unobserved.

Due to the small size of the fibre bundle there is little opportunity for a study of the spacing of the new cells. However, it is noticeable during dissection that there is generally an appreciable length along the fibre bundle, between the tips of cells in the same relative orientation, and that two ends together are rarely found.

Despite the bonding and lack of parallel arrangement in the bundle, the cells fit closely together, being separated from contact by a uniform middle lamella. This, although solidly in the center of the bundle, does not extend out uniformly to completely surround the outside cells.

The flax fibre bundle (Fig. 29) is also irregular in shape, but tends to be circular. The cells per bundle are between Manila and jute. At low magnifications the bundle is light in color, and the cells are slightly twisted as they lie together. Due to their comparatively long length, this twisting or out-of-parallel arrangement can impart an almost helical effect to the bundle if it is all in the same direction. At other times, when the twist differs, the effect is to open the bundle and expose the core. This condition is found more commonly in the smaller bundles and is best observed at low power. At high magnification, due to the smaller field, the cells appear to lie together and to be closely parallel.

The imperfections in the cells appear to occur together; this for both joints or nodes and fissures; and such an arrangement produces an almost perfect band around the bundle. At higher magnifications, randomly scattered small fissures are visible, and these to some extent reduce the orderly banding effect. This, however, still persists, and, although every cell is not proportionately affected, there is a decided belt of fissure across the bundle. The number of imperfections is not so great as in jute, but their size and severity are greater.

The same principle of carefully-spaced new cells is evident. Due to the longer length of the cells themselves, there are fewer tips per unit length of bundle, and a space, filled by full sized cells, is generally noticed between two tips. The cells really follow a randomly staggered pattern so that two tips rarely occur together.

These three diagrammatic drawings are intended to show the arrangement and relationship of the cells as they exist in the bundle; for it is in this form that they are used commercially. It must be remembered, however, that in bundle form considerable detail is hidden that can be observed in single cells and that the illustrations represent but a very small part of the length of the fibre bundle.

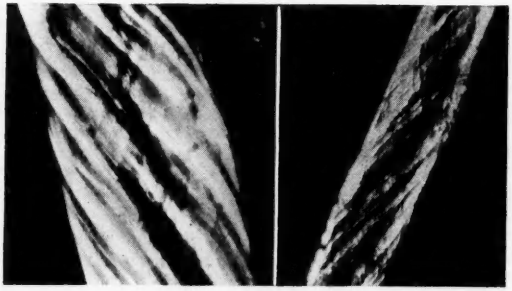


FIG. 30. FIG. 31.

FIG. 30. Flax cell swollen and compressed and viewed by plane polarized light between crossed nicols. Note the "S" spirality of the fibrils.

FIG. 31. A single jute cell in polarized light after swelling and slight compression. Note the "Z" spirality and the successive stages of disintegration to very nearly the ultimate fibrils in places.

There is undoubtedly a connection between cell arrangement and the strength and stretch of the commercial fibre. A random arrangement of the cells in the fibre bundle results in a much higher strength, both as to tension and flexure, than an orderly or systematic arrangement where the cells were placed tip to tip, or butt to butt. This also affects stretch; a much greater elongation before rupture is possible with a random arrangement of the cells, as they are able to slip by each other and still leave a continuous strand of cellulose throughout the bundle which would not be the case in an end-to-end arrangement, where slipping would cause a break in the length of the cellulose order.

The Camera-Lucida Drawings

It must be remembered that all of the camera-lucida drawings are subject to errors. No drawing is the exact duplicate of the image seen, and it is impossible to make it so. The personal equation is great enough in itself to prevent two drawings of the same specimen being entirely alike even under identical conditions, and, when to this is added the factors of change in illumination, focus and setting, the differences become more marked. It is only by using the utmost care in all departments of the set-up and nearly perfect specimens that results precise enough for comparison can be obtained. This is especially true when the object sketched is small, as in the case of the cross sections of the cell taken near the

tip. In such an instance it is the relative size and shape, not the definite outline, that must be considered.

In this connection, the occurrence of errors introduced into the object itself, due to preparatory techniques, should not be overlooked. The chief field here would seem to be in the preparation and cutting of cross-sections. Solvents or softeners for this purpose, as well as water, whether hot or cold, are known to have an effect on the fibre itself. Embedding mediums may have some effect due to their tenacity and compressive action; while the actual act of cutting is probably responsible for considerable changes in the shape and *arrangement of the cells in the fibre bundle.* Atmospheric changes also have an appreciable effect.

Discussion

The greatest clue to the structure of bast fibre cells is given by a study of their intimate composition, after suitable methods of displaying this have been employed. As there is a great degree of similarity concerning this structure among the bast fibres studied, in the following discussion the fibre referred to will always be flax unless specifically mentioned otherwise.

There is no doubt that the fibre cell is not homogeneous. A study of the cross-section, especially after staining, shows at least two distinct rings of layers in the central portion of the cell wall, and, although this part of the investigation has not been pushed extensively, on occasions more, even up to six or seven layers, have been observed. The use of an oil-immersion objective, even without staining, reveals two distinct structural layers other than the sub-division in the central portion; these are an outside wall or cuticle and a lumen sheath. The latter may not be an actual integral part of the cellulose of the cell, but a layer due to protoplasmic deposit. The difference observed is particularly noticeable when the light-transmitting ability of the section is considered. Thus, the middle portion can transmit light very well, despite the thickness of the section, while the other two thinner layers have not this ability to nearly such an extent.

Nodder [12] notes the previous findings and the likelihood of a radial structure in bast cells, resulting in a Brewster-cross effect; and in an earlier paper [6] reports a distinct and regular lamination of the cell wall; while the beading or strangulation effect produced by the viscose method of swelling, as reported by Herzog,[7] illustrates a difference in reactive power to this treatment between the layers. Such distinctions are probably due to, and are certainly best explained by, a difference in the micellar arrangement of the different layers. Searle,[13] by swelling the cross-section in NaOH and then subjecting it to pressure, has been able to differentiate as many as 50 separate layers, which may be analogous to the daily growth rings in cotton as reported by Balls.[14] However, to date, due to the lack of a definite datum from which to figure the time of development of a particular cell in the growth of the flax plant, there has been no positive correlation or proof of this.

When a longitudinal section of the cell is inspected, further extremely important facts concerning structure can be obtained. Nodder [12] reports that even in untreated fibres longitudinal separations in the cell wall can be observed, and that these, following the inclination of the fibrils, lie at

an angle of 10° or more with the fibre axis. In this study it has not been possible to see or measure these. What might sometimes be mistaken for them, have proven on more careful manipulation to be interference or diffraction bands, and there is a good possibility that these have confused the author mentioned. Such a possibility is emphasized after inspecting the diagrams and photographs of his earlier article,[6] where detail not found in this study is incorporated in the structural figures. However, it is positive that such detail can be clearly seen when the fibre is treated in a swelling medium and then compressed. Figs. 32, 33 and 34 show that at first the cell widens but little, and that the separations or dividing parts in the cell wall are small and close together. Later, the cell expands largely and the previously solid wall divides up into band-like formations which are spirally inclined. The band or spiral complex is in reality composed of numerous fibrils, almost below microscopic range in size, but which, by severe mechanical treatment, can be shown as extremely fine single filaments. Careful focusing on the upper side of a partially swollen fibre will show that these fibrils, in bands or as discrete units, run in "S" spirals for flax and "Z" spirals for jute and Manila.*

There has been some confusion as to the correct direction of the spirals in bast cells. Certain writers have concluded that there is a different direction of spirality at the same place in the same cell, but this has not been so found. Thus, Searle[13] describes a right-hand spirality for the fibrils in the inner layer of the cell and a left-hand spiral for those composing the outer layer. There is no doubt but that the appearance of the fibre as well as of the cell is deceiving, and seemingly a strong indication should be the direction of the drying twist, but careful focusing can also be used. The mistake is made when a slight change in the fine adjustment lowers the focus from the upper, outside surface of the cell (where the direction seen is correct) to some point further down in the body of the cell; for it must be remembered that the course of the spiral fibrillar structure follows the pattern of a barber's pole, and that "S" inclinations on the upper surface appear as "Z" inclinations when viewed on the opposite side through the specimen.† A further confusing effect is produced due to the difference in refractive index of the mounting medium and the cell, and, if the study is between crossed nicols, the extinction of one-half of the spiral bands upon rotation through a small angle on either side of the cross-hairs marking the planes of the nicols (as illustrated in the appendix) is most deceiving.

This study would indicate that the conception of two definite layers with different directions of spirality in the cell wall can be discarded. It was noticed, however, that there was a difference in the fibrils themselves.

* A further check is offered by the drying twist of the cells. A flax cell lifted from water into air will upon drying always turn in one direction. Thus, if the cell is held with the far end free, the turning, when viewed in this direction (the fixed end being nearer to the eye), will always be counter-clockwise.

† Again, looking at this error from the purely mechanical point of view of the mechanism of the fine focusing adjustment of the microscope: The smallest division on the dial for fine focusing represents a lowering of the objective approximately 2½ μ—this with the finest mechanism. Most microscopes are graduated to lower at approximately 5 μ per single division. The maximum thickness of a compressed cell averages 7 μ. Allowing nothing for the lumen we have a thickness of 3½ μ for one wall. If this is divided equally it means focusing must be to 1¾ μ at the maximum to precisely distinguish different structural details in one cell wall. This can be done, but only with the very greatest care. It is much more probable that distinction will take place between the upper and lower walls, rather than between the upper and lower halves of one wall.

FIG. 32. Short length of single Manila cell between crossed nicols. Note the fibrillar structure. On the upper surface the spirality is "Z" while on the under surface it is "S."

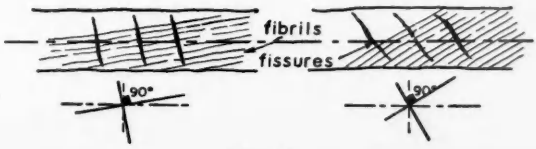


FIG. 33.

A B

FIG. 34. A.—Flax cell in position of parallel extinction between crossed nicols. B.—Fissures and markings in the approximate position of extinction. It should be noted that the portion of the cell which here appears black is actually red and orange in the original.

So far only a dimensional difference has been observed, but the explanation of much of the cell's actions probably lies here. It was revealed that a broad ring of fibrils, which lie closest to the lumen, are larger and coarser than those just inside the cuticle. The change from large to small on working outwards from the center of the cell does not appear to be gradual. There is only a small separate layer of random size fibrils in which the change is not sudden. The large fibrils appear to form the major portion of this part of the cell, or the whole cell for that matter. The ring of fibrils around the outer cell is distinctly smaller than this, while the intermediate layer, where the fibrils are not distinct as to size but appear to change indiscriminately, is extremely narrow. It is the finest fibrils that have the greatest tendency to flare out from the cell and become distinct entities; the coarser ones appear more firmly bound and retain their bonded structure longer.

In Fig. 32 it will be seen that at the outside edge of the cell, where the thickness is smallest, an apparent reversal in direction of spirality is shown. It is due, of course, to the depth of focus being sufficient to clearly show the top and bottom of the specimen at the same time. In like manner, the more open disintegration has left the cell, or the flatter compression has made it, the more chance there is for seemingly contradictory spirality to appear in the same focus.

Leaving the spiral structure of the cell for the time being, it would be well to fix attention on an even more discussed factor: namely, the fissuring or dislocation marks. The use of casts has shown that the majority of prominent fissures are capable of leaving an impression in the casting medium, and this in turn has been interpreted as an actual break or opening in the cell surface. By means of longitudinal split sections it was possible, in the case of ramie, to show that these open fissures also occurred inside the fibre, mainly branching out from the lumen,* and Searle [13] reports witnessing the development of transverse planes of segmentation extending outward from the lumen of a swollen flax cross-section. These fissures, or transverse planes of weakness, which have been described under various terminology throughout the literature, are particularly evident when the cell is between crossed nicols. The longitudinal views of single cell sections, and the diagrammatic representation of fibre bundles, besides the photomicrographs, give an excellent idea of the appearance of the cell and these transverse markings. However, when a normal cell is subjected to the treatment designed to show its spiro-fibrillar structure, there is a strong tendency for these transverse markings to disappear. If the same treatment is given to a cell previously subjected to carbonizing action (acid soaking, followed by quick drying) the results are very different. In the latter case the swelling and compression cause the fissures to become decidedly more pronounced and eventually to completely cross the cell, dividing it into two parts. The resulting ends are rough and jagged and lie at other than a right-angle to the longitudinal axis of the cell. Sometimes under this treatment other markings will appear in the cell very similar to the fissures themselves. They generally run from the lumen outward, but, except for this slight indication, there is no means of visual identification. Pressure applied above them, however, causes their disappearance, and it is possible that they are merely diffraction effects caused by the breakdown of the normal cell structure

* Osborne. *Tex. Rsch.*, V, 2, 77 (1934).

due to swelling. Certainly, as Searle [13] has observed, there must be a distinction drawn between the two types of fibre markings, only one of which is capable of developing into an open-cleavage plane.

The angle that the fissures make with the cell axis, together with the fact that all fissures angle in the same direction on one surface, point to a connection between fissures and fibrils, rather than fissures and the cell itself. If two groups of fissures, lying at different angles to the cell axis, are observed, and the specimen is then treated by NaOH and pressure, the portion in which the fissures make the greater angle with the cell axis will show fibrils at a smaller angle with the cell axis; while, where the fissures made a smaller angle, the angle of the fibrils will be greater. The average angle between fissures and cell axis, as measured by a filar micrometer and graduated, rotating stage, is 81°, and, from measurements of the casts, not more than 4 or 5° less than a right-angle. In any event, it seems seldom to be less than 70° and approaches, and often is, truly perpendicular. The angle that the fibrils make with the cell axis, after as little treatment as possible to make them evident, averages 15° with a variation of $\pm$ 4°.* This figure is in accord with findings of other writers, but it must be remembered that it was taken from a distorted fibre, and, if the hypothesis to be advanced is sound, the average angle would be considerably smaller.

Before proceeding further, however, it might be well to illustrate the angular relationship discussed by means of the diagrams of Fig. 33. This shows that the relationship between fissures and fibrils is such as to tend to keep the angle between them a right-angle, and that the fissures in this respect are similar to the slip-planes in cotton, described by Balls.[14] Additional information can be thrown on this aspect of the subject from a consideration of the cells' reaction in polarized light, which follows:

All textile fibres are doubly refracting and flax is one of the strongest in this respect. Between crossed nicols the body of the cells gives complete parallel extinction. (Fig. 36.) This is also true of the fibre bundle, although, since here there is a slight difference in cell alignment, the effect is not so good. It is also possible to show that the fibrils themselves (after the cell is swollen) give parallel extinction; and, although it is difficult to get a single fibril in a long, straight length, such has been found. Otherwise, if the fibrils are separate, but curved, a black extinction band will pass down their length as different units of it in turn become parallel to the planes of the nicols. Such bands are visible in Fig. 35. With complete extinction at exactly parallel positions the fissures show up as brightly lighted lines. As the fibre, cell, or fibril, is moved from this position it gains in color up to the 45° line, where it is at its maximum brilliance. During this course (Fig. 34) the fissures lose their prominence and the striking contrast is lost, but it is possible to see that many of them become extinct and bright again as they pass through a parallel position.

It would be interesting to see just exactly what is the reaction of the fissures. This has been accomplished by using high magnification—approximately 1800 diameters. First, by careful focussing on the edge of

* Contrary to a generally accepted view, the fibrils in ramie have been found to lie at a slight angle with the horizontal axis of the fibre. This angle varied from almost immeasurably small amounts to as much as 6°. The measurements were made from camera-lucida drawings, but the fibre was merely water-softened and only slightly compressed. The fissure angles also substantiate the non-parallel arrangement of the fibrils and fibre axis, in many cases varying 4 to 5° from a right-angle relationship.

the cell, it is possible to see the actual fissure break into the wall. That this is an actual opening is shown by the fact that, on rotation, whether alone between crossed nicols or with 1st order, or quarter-wave plate, the opening will not show other than the color of the background. Such breaks are rarely found; the usual ones do not allow the background to be seen through them, as is only possible when the break through the wall is parallel to the line of sight. These others, however, show a parallel extinction, turning completely black between crossed nicols alone, magenta with the first selenite, or green-yellow with the quarter-wave plate; the latter being the most difficult to observe. In the fibre bundle, cell or fibril, com-

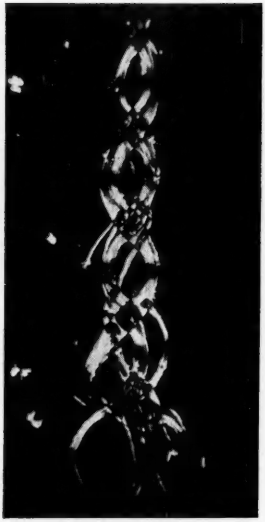


FIG. 35. Jute cell parallel to plane of polarization between crossed nicols.

plete extinction occurs only when exact parallelization is reached. A 1° variation from this is noticeable. The possible variation with the fissures is much greater; they appear completely extinct for a range as great as 7° on each side of the parallel position. Much of the explanation concerning this and the other observable phenomena, and its interpretation, must be tempered by a realization of the many and complex structures these fissures can assume.*

The optical sign of the cell wall and the character of the indicatrix have been discussed by various workers. Herzog [7] gives an average figure for refractive index of 1.56 and .067 for specific birefringence. The fibrils are optically positive; *i.e.*, light travels with the lowest velocity in the fibre when its vibrations are parallel to the fibril axis. Nodder [12] states that the

* Osborne. *Tex. Rsch.*, V, 2, 84 (1934).

use of a selenite with cross-sections shows that the shortest axis of the indicatrix is radial. The longest axis is parallel to the fibrils, and the intermediate axis lies tangentially and at right-angles to the fibrils.

A phenomenon interesting on two counts (its relation to the fissures and its bearing on structure) was observed during an endeavor to obtain chemical cross-sections from single cells. After single cells were dissected they were thoroughly carbonized by boiling in 10% H_2SO_4 and drying in a conditioning oven. They were then soaked in a 10% caustic solution and compressed and jarred after the procedure of Kelaney and Searle.[15] It was quite easy to separate discrete fibrils from the cell, and, although these were noticeably changed, they still showed identical reactions in polarized light to a normal sample. Agitation, so as to produce wave motion in the sample as described in the reference,[15] will result in the fibre bundle or cell segmenting into thin cross-sections perfectly transverse to the longitudinal axis and very regularly spaced at intervals of approximately 10 μ. If the manipulatory technique is not correct for this formation the sample splits both longitudinally and transversely, and, if continued, the entire sample will break down into a multitude of small rod-like units. These can be divided again and again until they become almost dust-like, but the interesting points are that, up to complete pulverization, they show the same reactions between crossed nicols as the normal cell, and that their derivation from the spiral component of the cell is by a process very similar to cleavage.

There does not seem to be a direct relationship between these transverse section planes and the much larger, easily visible fissures. The former are regularly spaced, very large in number and lie at right-angles to the longitudinal axis of the sample; while the fissures themselves are few in number, scattered at random, or grouped, and lie at right-angles to the spiro-fibrils of the sample.

Conclusions

From the purely physical point of view these findings lend further weight to the two-phase character of vegetable fibres: the combination of both colloidal and crystalline properties. So much work has been done on the nature of cellulose that its micelle arrangement and crystalline structure are as well known as those of any other substance.[16] There has, however, been the contention that the double refraction exhibited by textile fibres in polarized light, and by flax in particular, was due to internal stresses and strains achieved during growth or subsequent operations, and not to the structural arrangement of the object. That this property is retained as fully as ever after the fibre has been divided into discrete entities, soaked in water, expanded in swelling media, and even ground to minute fragments (when, seemingly, strain conditions could be relieved or modified) is strong evidence that there is a definite arrangement and a crystalline structure.

If the fibrils themselves are considered as crystalline growths or crystalline aggregates and there is a definite relationship between them and the fissures, these latter may be regarded as twinning or gliding planes, similar to those produced in minerals (mica, calcite) by pressure; a view that has been expressed by other workers. This, however, does not explain the transverse section plane produced by carbonizing, swelling, and compression.

It seems more likely that the cause of weakness in the cell or fibre bundle may be explained in the same manner as for ramie.* Here it was pointed out that deep, complementary fissures greatly reduced the area of section to be broken; that the high concentration of stress at the end of an open crevice, when the elastic limit is quickly reached, results in a tearing rupture at lower load than straight tension, and that failure by bending or flexure is also strongly induced. Furthermore, analogous to Balls'[14] theory of mechanical damage and slip-planes in cotton, Muller[11] has shown that severe mechanical treatment can produce large numbers of fissures in flax, while at the same time reducing the strength of the fibre.

The local lateral separations of the fibrils near a fissure mark, best detected by differential staining, would not have as great a weakening effect as the one just described, although it admittedly tends to reduce the cell's strength. Furthermore, it seems extremely unlikely that a loss in strength is due to slippage between the fibrils themselves, as fibres in yarns can and do slip, for, although the fibrils can be separated as units, they are strongly bonded together and are more or less continuous in length.

Differential staining at the fissures might also indicate a chemical difference here. The theory has been advanced that at these points, which are admittedly points of weakness, there has been a depolymerization of the cellulose, and that, therefore, it is more susceptible to attack by hydrolysis or oxidation. (Practically, however, it seems improbable that the fibre would normally be subjected to a *chemical* action severe enough to bring about depolymerization.) Searle[13] states that natural weakening through tendering is brought about by either of these chemical reactions, and that, though weakness does not necessarily immediately follow fissure formation, it is always a potential point for chemical attack.

This does not seem to be the time to point to any specific conclusions. There is evidence that points to each contention as being correct. Actually, the result is probably due to all, although perhaps not equally to each. However, in view of the fact that the fissures are actual openings in the cell wall, some change in the current theory must be made; while if these breaks could be found in the fresh fibre uninfluenced by any chemical action a strong weight would be placed in favor of the purely mechanical imperfection idea of weakness.

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Corrections

The following corrections were received from Dr. Mearl A. Kise, author of the article on "Silk Fibroin Dispergation," appearing in the July number, pp. 401–14, too late to be made:

P. 406. In lines 14 and 15, change cc. to ml.

P. 408. In second column of Table 1, fifth line of figures, change 37.25 to 47.25. In same table change heading of fourth column from d^{25° to $(\alpha)_D^{25^\circ}$

Degradation of Silk Fibroin by Acid and Alkali

By EUNICE CHAMBERLIN WALDE and RACHEL EDGAR *

Silk is frequently subjected to acid or alkaline solutions during fabrication and use. This paper presents a résumé of the meager literature of partially hydrolyzed fibroin, and a quantitative description of the effect of the concentration of the hydrolytic agent on the course of the acid and alkaline degradation of silk fibroin and wild silk fibroin as followed by weight, nitrogen, wet warp breaking strength, and elongation at breaking load of the residual fibroin.

ACIDS and alkalies have been common reagents in the technology of silk since the beginning of its use as a textile. Silk may be treated with acid in the purification, hardening, bleaching, or degumming of raw silk; in mordanting, weighting, dyeing, stripping, felting, crêping, scrooping, lustering; production of transparent and preservative finishes, or upon exposure to carbonization, acid fumes, or sweat. Some alkaline treatments of silk are reeling, softening, degumming, fixing and removal of mordant, bleaching, dyeing and printing with developed sulfur, or vat dyes, delustering, cleaning, production of transparent or preservative finishes, and exposure to mercerizing baths, sweat, and ammonia. The greater resistance to acid and alkali of wild silk fibroin, compared with silk fibroin, has been made use of in degumming and in distinguishing between the two silks.[27, 3]

Acid Degradation of Fibroin

Development of methods for the quantitative estimation of silk [42, 16, 17, 14, 26, 34, 12, 8] and prevention of deterioration of silk [15, 25] have produced some scattered data of the effect of acid on the weight, nitrogen, and mechanical failure of fibroin. It has been noted that dilute acid and alkali cause silk fibre to separate into fibrils,[27] that acid increases the instability of fibroin to light,[22] has no effect on the X-ray diagram of fibroin, and brings about a slower mechanical failure than occurs with alkali.[33] The isoelectric range of silk fibroin has been reported as pH = 1.3 to 5.24,[40, 6, 10, 36, 24, 23, 21, 7, 9, 40, 30] and that of wild silk fibroin as pH = 2.6 to 2.79.[4, 5] It has been suggested that the swelling of fibroin in 2 *N* or more concentrated hydrochloric acid, not increased by pretreatment with acid, may be caused by structural disintegration.[33] The hydrolysis of dissolved fibroin, more rapid than that of casein and slower than that of gelatin, has been shown more rapid the higher the concentration of acid, although slower in acid than in alkali.[28] The acid hydrolysis of silk fibroin, as measured by increase of amino nitrogen, has been described as a reaction of the second order.[18]

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Alkaline Degradation of Fibroin

Methods for the extraction of nitrogenous finishing substances record some effects of dilute alkaline solutions on the nitrogen [11] and weight [13] of fibroin. The mechanical failure of fibroin caused by alkali in degumming, as measured by breaking strength and elongation at breaking load, has been shown to increase with increasing time, temperature, and concentration of alkali.[20, 2, 27, 29, 44, 45, 39, 43, 46] Alkali-treated fibroin has been reported more stable to light [22] but less stable to weighting [38] and sulfur dioxide.[31] It has been suggested that imbibitional swelling of fibroin may begin in alkali by the binding of water at the carbonyl group of one peptide chain and the imino group of an adjacent parallel chain upon the non-reversible opening of a co-ordinate linkage; further swelling has been ascribed to structural disintegration.[32, 33] It has been shown that fibroin is more readily attacked by alkali than by acid, that pretreatment with alkali increases the swelling of fibroin, and that this swelling is proportional to the concentration of sodium hydroxide between 0.01 *N* and 6 *N*.[23]

Although the hydrolysis of silk fibroin has been extensively studied in attacking the problem of its structure, few quantitative data of the residual fibroin have been published. To measure the effect of the concentration of the hydrolytic agent we have treated a plain-woven fabric of silk fibroin and one of wild silk fibroin with acid for 10 hours at 40° C. and one hour at 100° C.; with alkali for 10 hours at 25° C. and 40° C. and one hour at 100° C., and determined the weight, nitrogen, wet warp breaking strength, and elongation at breaking load of the residual fabric. The effect of concentrated acid and alkali on the silks at 15° C. has been measured in the same way. A wider range of concentrations of acid and alkali has been studied than is commonly encountered in the processing and use of silk; low concentrations as those of degumming, bleaching, dyeing, scrooping, service and maintenance; and high concentrations as recommended for producing crêpe effects and transparent finishes.

Experimental Procedure

Preparation of Fibroin.—Silk crêpe, plain-woven in the gum, was boiled one hour in 100 volumes of 10% solution of neutral olive-oil soap and rinsed; after a second repetition, the fabric was rinsed in boiling water three times, dried, and cut into samples for analysis. The wild silk fibroin (0.60% ash) was prepared by boiling plain-woven pongee in 100 volumes of water one hour and then rinsing and drying the fabric. The silk samples were extracted 18 hours with anhydrous ether in a modified Soxhlet apparatus. Five-gram samples of the ether-extracted degummed silk (0.23% ash) were dried to constant weight (within ± 0.008% by the method of tares in seven one-hour periods) at 105° C., plunged into boiling water and again dried to constant weight to determine the ether retained by the fibroin. A constant retention of 0.14%, compared to a reported 1.5%,[35] has been disregarded.

Acid and Alkaline Degradation.—Two hundred cc. of water, hydrochloric acid (standardized by conversion of the chloride into silver chloride, which was weighed), or standard sodium hydroxide were measured into a 250-cc. test-tube fitted with a rubber stopper and placed in a water bath at 25° ± 0.1° C., or 40° ± 0.1° C., until the liquid attained the temperature

TABLE 1

EFFECT OF CONCENTRATED ACID AND ALKALI ON SILK FIBROIN AT 15° C.

Silk	Treatment	Time Minutes	Residual silk fibroin		Breaking strength of wet warp lbs. per in.	Elongation at breaking load %
			Weight % of silk	Nitrogen % of silk		
Silk fibroin	(a) None		100.0	18.67	44	29
	(b) Hydrochloric acid, sp. gr. 1.145	0.5	85.3	16.02	<1	
	(c) Sodium hydroxide, sp. gr. 1.410	5.0	99.9	18.57	34	23
Wild silk fibroin	(a) None		100.0	18.27	16	23
	(b) Hydrochloric acid, sp. gr. 1.145	0.5	99.1	18.15	16	25
	(c) Sodium hydroxide, sp. gr. 1.410	5.0	99.5	18.22	11	16

of the bath. Approximately five grams of silk fabric, dried to constant weight (± 0.0004 g.) at 105° C., or 10 breaking strength specimens were then submerged in the liquid for 10 hours, removed, and thoroughly washed until the rinse from the acid-treated silks showed no opalescence with silver nitrate and that from the alkali-treated no color with phenolphthalein.

Tests at 100° C. for one hour were made similarly in a 500-cc. balloon flask fitted with a water-cooled condenser and heated over a boiling water bath. Titration of blank determinations showed no concentration of the test solutions upon boiling.

In preparation for treatment with concentrated acid or alkali the silks were immersed in water and then centrifuged for a minute. The silk sample was then gently stirred into a 100-cc. portion of the test solution at 15° ± 0.1° C. and, upon removal, was vigorously stirred in successive liter-portions of water until the rinse gave no test for chloride or alkali. Upon treatment with hydrochloric acid, sp. gr. 1.145, for 30 seconds, silk fibroin shrank and became sticky while wet so that rinsing free of chloride was difficult. Both silk fibroins became transparent after five minutes in sodium hydroxide, sp. gr. 1.410, but assumed their usual appearance when rinsed.

Weight, Nitrogen, Wet Warp Breaking Strength, and Elongation at Breaking Load.—The five-gram samples of silk were dried to constant weight and analyzed for nitrogen by the Kjeldahl-Gunning-Arnold method;[19] the one-inch warp strips were tested wet at once for breaking strength and elongation at breaking load by means of a Scott universal tester and autographic recorder.[1] Values reported for weight and nitrogen are the averages of four or more closely agreeing determinations, and values for breaking strength and elongation at breaking load are the averages of ten determinations.

The greater degradation of fibroin by alkali, than by acid, is shown in Figs. 1 to 6.* Table 1 shows the effect of concentrated acid and alkali on the two silks at 15° C.

* The quantitative data may be had by addressing the editor.

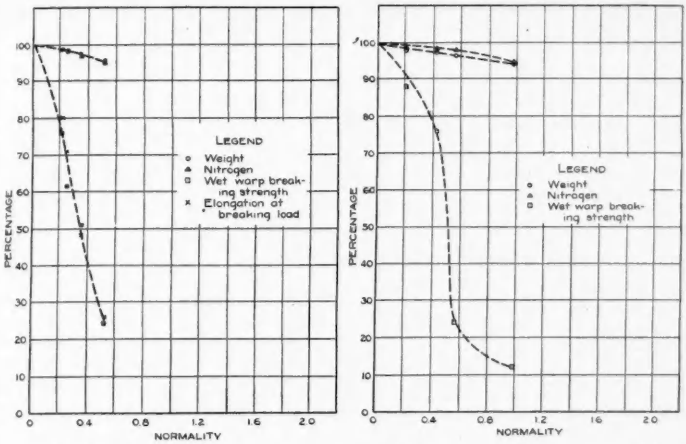

FIG. 1. Effect of sodium hydroxide in 10 hours at 25° C. on the weight, nitrogen, wet warp breaking strength, and elongation at breaking load of a plain-woven silk fabric.

FIG. 2. Effect of sodium hydroxide in 10 hours at 25° C. on the weight, nitrogen, and wet warp breaking strength of a plain-woven wild silk fabric.

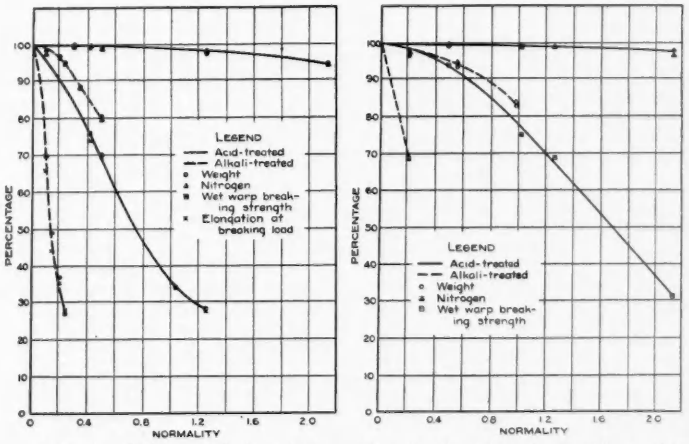

FIG. 3. Effect of hydrochloric acid and of sodium hydroxide in 10 hours at 40° C. on the weight, nitrogen, wet warp breaking strength, and elongation at breaking load of a plain-woven silk fabric.

FIG. 4. Effect of hydrochloric acid and of sodium hydroxide in 10 hours at 40° C. on the weight, nitrogen, and wet warp breaking strength of a plain woven wild silk fabric.

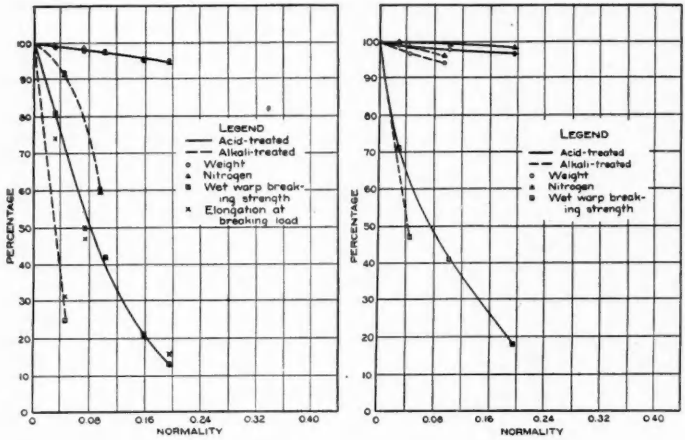

FIG. 5. Effect of hydrochloric acid and of sodium hydroxide in one hour at 100° C. on the weight, nitrogen, wet warp breaking strength, and elongation at breaking load of a plain-woven silk fabric.

FIG. 6. Effect of a hydrochloric acid and of sodium hydroxide in one hour at 100° C. on the weight, nitrogen, and wet warp breaking strength of a plain-woven wild silk fabric.

Alkaline degradation of silk fibroin tends to leave a residue poorer in nitrogen, and acid degradation a residue richer in nitrogen than the original fibroin. Alkaline degradation of wild silk fibroin leaves a residue richer in nitrogen than the original fibroin or the residue from acid degradation. The more rapid loss of tensile strength than of weight or nitrogen indicates that acid and alkali first bring about a general disintegration of fibrous structure without the formation of soluble products.

Summary

1. The course of the degradation of silk fibroin and wild silk fibroin, in alkali at 25° C. for 10 hours, and in acid and in alkali at 40° C. for 10 hours and at 100° C. for one hour, has been followed by determination of the weight, nitrogen, and mechanical failure of the residual fibroin.

2. The effect on the silks of hydrochloric acid, sp. gr. 1.145, in 30 seconds and sodium hydroxide, sp. gr. 1.410, in five minutes at 15° C. has been measured in the same way.

3. Weight and nitrogen of fibroin have been shown to decrease very similarly and to be logarithmic functions, $y = ax^b$, of the concentration of the hydrolytic agent.

4. The greater effect of alkali as compared with acid, the greater mechanical failure as compared with loss of weight or nitrogen, the effect of temperature, and the greater resistance to acid or alkali of wild silk

fibroin compared with silk fibroin have been quantitatively described for the degradation of the two silk fibroins.

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Abstracts

This section is compiled under the direction of the Committee on Abstracts and Bibliography: Prof. E. R. Schwarz, Chairman, Dr. W. E. Emley, Prof. Louis A. Olney and M. W. Weiss.

I. Fibres (Synthetic and Natural) and Fibre Analysis

Flax Retting. R. Cerighelli. *Agron. Col.*, 1934, V. 23, P. 33–9; *Chim. et Ind.*, V. 33, P. 442.

From a comparison of natural, phys. (treating with boiling water, or with steam under pressure at 110°), and chem. (boiling with soap or Na_2CO_3 solns. at atm. pressure or under pressure at 110°) retting showed that natural retting is the most economical and yields the highest-grade product. (Copied complete from *Chem. Abs.*, 1935, V. 29, Col. 3167.) (W)

Flax: New Process for Retting. G. O. Searle. *Tiba*, 1935, V. 13, P. 79.

The essential feature of the process consists in maintaining a uniform temp. throughout the mass of liquid by a rapid circulation of the liquor inside the flax bundles, which are preferably piled vertically. (Copied complete from *Chem. Abs.*, 1935, V. 29, Col. 3168.) (W)

Flax and Hemp: Influence of Iron and Other Metals in the Microbiological Retting of. IV. D. Carbone and A. Moggi. *Boll. soc. intern. microbiol.*, Sez. ital., 1935, V. 7, P. 30–5; *Chem. Abs.*, 1934, V. 28, Col. 2909.

Cu, Zn, Pb and Fe exert an injurious action in the microbiol. retting of flax and hemp; Cu, Fe and Pb even affect the color of the fibres, which become dark green or gray. Al is harmless. Sn, which does not act on the color, hinders the anaerobic retting of hemp, but not the aerobic retting of hemp or either retting of flax. (Copied complete from *Chem. Abs.*, 1935, V. 29, Col. 3168.) (W)

Keratin: Structure of. Maurice Déribéré. *Tiba*, 1934, V. 12, P. 905–10; *Chem. Abs.*, 1935, V. 29, Col. 3168. (W)

Halogenation of Wool. Charles Courtot and Alfred Baron. *Compt. rend.*, 1935, V. 200, P. 675–6.

The treatment of wool with Cl or Br causes marked morphological changes while I is practically inactive although definite adsorption occurs. Long exposure to an excess of Cl or Br but not I in H_2O causes 60% of the wool to dissolve. Oxidation of S does not affect the soly. Soln. by chlorination or bromination produces a change in the polarization of light.

On steam distn. the neutralized soln. yields a mixt. of aldehydes. Thoroughly washed chlorinated wool has an oxidizing power of 30 times that of the Cl present. (Copied complete from *Chem. Abs.*, 1935, V. 29, Col. 3169.) (W)

MILDEW OF WOOL. L. Armand. *Tiba*, 1934, V. 12, P. 675–9, 751–5, 831–5, 917–23; 1935, V. 13, P. 23–7; *Chem. Abs.*, 1935, V. 29, Col. 3169.

A report on numerous tests, extending over a period of two years, on the inhibiting action of dyes toward the mildew of wool. (W)

II. YARNS AND FABRICS

FIRE-RESISTANT DOPED FABRIC FOR AIRCRAFT. Gordon M. Kline. *B. S. J. Rsch.*, May, 1935, P. 575–87.

Cellulose nitrate dope now commonly used to cover the fabric on the wings and fuselage of airplanes is very flammable and its replacement by a less-hazardous product is desirable. The development of a satisfactory nonflammable dope by the use of natural and synthetic resins or mixtures of synthetic resins with cellulose nitrate and cellulose acetate has not proved to be feasible. An airplane covering with very good resistance to ignition may be obtained by the application of a 3:7 boric-acid-borax mixture to airplane cloth and subsequently doping it with cellulose acetate. Cellulose acetate dope is now only moderately more expensive than cellulose nitrate dope. The cost difference becomes insignificant when the lower fire hazard resulting from the use of the much less flammable cellulose acetate dope is considered. (C)

LATEX: APPLICATION OF. John F. Hotchkiss. *Tex. Wld.*, 1935, V. 85, P. 666–7; *Chem. Abs.*, 1935, V. 29, Col. 2023, 3191.

The compounding of latex for backing for pile fabrics and its application to the fabric are described. A coagulation test, pH detn., viscosity measurement and evapn. test are suggested as control tests. (W)

RUBBER: APPLICATION OF—TO RAYON YARNS AND FABRICS. H. Roche. *Silk J.*, 1934, V. 10, No. 119, P. 14; *Chem. Abs.*, 1934, V. 28, Col. 4241.

Rubber latex, stabilized by the addn. of casein, gelatin or glue, can be mixed with viscose soln. and emulsified to give a spinnable mixt. contg. 2–4% of latex, by wt. The rubber can be vulcanized with SCl_2 dissolved in CS_2 or the S from the viscose can be used, with an accelerator. Rayon fabrics coated with a mixt. of viscose and rubber can be treated with an acidic gas, such as SO_2 or AcOH vapor. The elasticity of rayon knitted fabrics is improved by stretching in the wet state, drying, applying a sheet of rubber to one surface and vulcanizing. Waterproof fabric is produced by applying a layer of a rubber compd. to the fabric base; this is followed by a second layer consisting of a mixt. of a cellulose deriv. and a miscible synthetic resin. Synthetic (acetate) yarn materials are coated on both sides with a soln. of rubber, vulcanizer, S and an accelerator; they are then vulcanized at 100°. Dyeing and delustering rayon and synthetic yarn materials with latex and colored or white pigments are mentioned. (Copied complete from *Chem. Abs.*, 1935, V. 29, Col. 3170.) (W)

SIZING RAYON: NEW METHOD FOR UTILIZING STRONG BONE GLUE FOR. Charles Seyd. *Rusta,* 1934, V. 9, P. 831, 833.

Attention is drawn to the advantages of glycerolated strong bone glue for sizing acetate rayon. (Copied complete from *Chem. Abs.,* 1935, V. 29, Col. 3170.) (W)

III. CHEMICAL AND OTHER PROCESSING (NOT OTHERWISE CLASSIFIED)

DYEING TULA FIBRE SHINY BLACK. A. Fritsch. *Z. ges. Textil-Ind.,* 1934, V. 37,.P. 575; *Chem. Abs.,* 1935, V. 29, Col. 1988. (W)

DYEING VISCOSE. N. N. Malyutin and D. Tz. Kantor. *Iskusstvenoe Volokno* (*Artificial Fiber*), 1934, V. 5, No. 7, P. 52–8.

Substantive dyes of the aniline blue type produced poor results. The S dyes gave good dyeings. (Copied complete from *Chem. Abs.,* 1935, V. 29, Col. 2361.) (W)

DYEING WITH NAPHTHOL AS. Carlos Gattiker. *Raca,* 1932, V. 1, P. 7–8; *Anales asoc. quím. Argentina,* V. 22, P. 230B; *Chem. Abs.,* 1935, V. 29, Col. 2749. (W)

DYES: ACTION OF HOT CONCENTRATED SULFURIC ACID ON. R. B. Forster. *J. Soc. Chem. Ind.,* 1934, V. 53, P. 384T.

The color of a soln. of dye in cold coned. H_2SO_4 may or may not discharge on heating. The effect obtained is of assistance in assigning dyes to their resp. classes. (Copied complete from *Chem. Abs.,* 1935, V. 29, Col. 1642.) (W)

FAULTS IN WOOL FABRICS: CHEMICAL INVESTIGATION OF. H. R. Hirst. *Tex. Mfr.,* Jan. 1935, P. 32–3.

The article is an abstract of a paper read to the Bradford Textile Society and discusses the principal faults resulting from chemical damage to wool or staining, together with general methods of avoiding unevenness of dyeing and stains. (S)

FREE ALKALIS AND THE DETERMINATION OF THE HYDROLYTICALLY DISSOCIATED ALKALI IN SOAPS: METHODS OF DETERMINING. Dr. J. Davidson. *Rayon and Mell. Tex. Mo.,* Jan. 1935, P. 55–6.

This is part one of a series of two articles and the methods used to determine the free alkali alone, without consideration of the hydrolytically dissociated alkali discussed. In the second article which appears in the *Rayon and Mell. Tex. Mo. for Feb. 1935,* P. 43, new research regarding the hydrolytically dissociated alkali and the importance of it in the soap are noted. (S)

FUROLE DYESTUFFS: FURTHER STUDIES IN THE. Ossian Aschan and Arthur Schwalbe. *Finska. Kemitsam. Medd.,* 1934, V. 43, P. 48–54; *Chem. Abs.,* 1935, V. 29, Col. 1987. (W)

Impregnating with Cuprammonium. Gustav Durst. *Kunststoffe*, 1934, V. 24, P. 197–8, 285–6; cf. *Chem. Abs.*, 1934, V. 28, Col. 2521.

A discussion of methods, difficulties and advantages of impregnating textiles with cuprammonium soln. prepd. from either Cu turnings, or pptd. Cu carbonate or green Cu oxide. (Copied complete from *Chem. Abs.*, 1935, V. 29, Col. 1990.) (W)

Indanthrene and Indigosol Dyes: Innovations in the Field of. K. Stierwalt. *Monatschr. Textil-Ind.*, Trade issue III, 1934, V. 49, P. 56–7; *Chem. Abs.*, 1935, V. 29, Col. 1641.

Several new dyes found to be very fast to light are mentioned. (W)

Indigosol Dyes for Dyeing Cotton. Georg Rudolph. *Z. ges. Textil-Ind.*, 1935, V. 38, P. 16–17; *Chem. Abs.*, 1935, V. 29, Col. 1988. (W)

Kier Boiling. John Bothameley. *Tex. Bull.*, 1934, V. 46, P. 6, 18.

A discussion of the prepn. of cotton piece goods prior to kier boiling and of kier boiling preparatory to Cl bleaching. (Copied complete from *Chem. Abs.*, 1935, V. 29, Col. 349.) (W)

Kinetics of the Acetylation of Cellulose Fibres. IV. Kinetics of the Acetylation of Ramie in the Fibre Bond, Steeped in Acetic Acid, and the X-ray Diagram of Triacetylcellulose. I. Sakurada and M. Shojino. *J. Soc. Chem. Ind., Japan* 37, Suppl. binding, 1934, P. 599–603; cf. *Chem. Abs.*, 1933, V. 27, P. 3814; 1935, V. 29, Col. 1254. (W)

Mordanting of Silk with Chrome Alum. IV–V. K. Hishiyama and M. Nakamura. *J. Soc. Chem. Ind., Japan* 37, 1934, Suppl. binding, P. 624–5; cf. *Chem. Abs.*, 1932, V. 26, P. 5761; 1935, V. 29, Col. 1254. (W)

Oil and Oil Spots. Wilhelm Bauer. *Monatschr. Textil-Ind.*, 1934, V. 49, P. 252–3.

Mixts. contg. 40–67% of mineral oil and 60–33% of rape-seed oil can be used in pretreating textile raw materials for bleaching and dyeing purposes as these mixts. can be removed by washing. Paraffin-free and decolorized mineral oil should be used as it disappears readily during the normal bleaching process. On testing mineral oil spots it was shown that the various grades differ in their resistance to washing out or emulsification. The mineral oil should also be free from H_2SO_4 (from the refining operation) which may cause trouble during bleaching. Difficulties may be experienced with oil spots which are caused by dirt or rust. They cannot be removed by washing but must be subjected to a special chem. treatment. Therefore, the occurrence of oil spots caused by excessive oiling must be avoided as much as possible in spinning, bleaching and weaving operations. Even if the textile should not be bleached at all any oil spots that appear should be removed with hot water and a suitable emulsifying soap. If such soap is not available or the removal of spots causes difficulty they can be pretreated with a slight amt. (1/5–1/10%) of one of the numerous fat solvents. (Copied complete from *Chem. Abs.*, 1935, V. 29, Col. 1644.) (W)

OXIDIZING AGENTS AND VAT-DYED COTTON. H. A. Turner, G. M. Nabar and F. Scholefield. *Nature*, 1935, V. 135, P. 68.

At pH 3–11 the e. m. f. of a Pt electrode in dil. NaOCl parallels the action of the soln. on pure cotton cellulose dyed with reduced vat dyestuffs. The latter is detd. from the viscosity of solns. of the fibre in cuprammonium hydroxide. (Copied complete from *Chem. Abs.*, 1935, V. 29, Col. 2363.) (W)

PRINTING ON UNCHLORINATED SILK WITHOUT SPECIAL HUMIDIFICATION AND WITHOUT YELLOWING THE WHITE IN STEAMING. Ed. Justin-Mueller. *Bull. soc. ind. Rouen*, 1934, V. 62, P. 345–7.

Silk can be printed without chlorination if $NaHSO_3$ is added to the printing color. There are a no. of colors which are not harmed by such addn. Tests on several colors (chiefly alizarin derivs.) showed that best results occurred with 135 g. of $NaHSO_3$ per 1000 g. of bath (bath contg. 27 g. of dye per 1000 g.). The colors contg. $NaHSO_3$ are humidified sufficiently in steaming, without a previous humidification. It is believed that the $NaHSO_3$ acts on the silk to produce albuminoids. (Copied complete from *Chem. Abs.*, 1935, V. 29, Col. 2361.) (W)

PRINTING WITH SOLEDON COLORS. R. C. Unsworth. *Dyer*, 1934, V. 72, P. 434.

The Soledon colors are the water-sol. esters of anthraquinone vat colors. The nitrite and steaming processes for applying them are described. (Copied complete from *Chem. Abs.*, 1935, V. 29, Col. 2361.) (W)

PHOSPHATE PRODUCT: NEW—FOR THE TEXTILE INDUSTRY. D. K. Manners. *Dyer*, 1934, V. 72, P. 385, 387.

Na metaphosphate, commercially known as "Calgon," softens hard water without the formation of sludge or ppt. This property makes it a helpful assistant in many textile wet processes, such as scouring and dyeing of wool; degumming, dyeing, and finishing of silk; and kier-boiling of cotton. (Copied complete from *Chem. Abs.*, 1935, V. 29, Col. 2362.) (W)

REVIVIFYING USED BOIL-OFF LIQUOR FOR ADDITIONAL DEGUMMING PURPOSES. H. H. Mosher. *Rayon and Mell. Tex. Mo.*, April, 1935, P. 70–73.

A brief article indicating how used boil-off liquors can be freshened up for further use and noting certain process details in connection with this treatment. It is stated that degumming solutions as conventionally employed are useful in stripping three or at most four lots of raw silk, further, that the exhaustion of such baths is due to a lack of alkaline potency in the liquor with a subsequent loss of fat dispersing power and the development of insoluble soap rather than a loss of the detergent. It is further stated that such solutions can be revivified by controlled alkaline addition by regeneration of the active soaps from insoluble lime and magnesium fatty acid complexes. It is stated that by such treatment the life of a degumming solution can be doubled with the resulting saving in chemical costs of something on the order of 50%. (S)

IV. Research Methods and Apparatus

Electrical Resistance of Yarns and Cloths: Measurement of. Variation in the Resistance of Silk with pH. W. S. Denham, E. A. Hutton and T. Lonsdale. *Trans. Faraday Soc.*, 1935, V. 31, Pt. 3, P. 511–19.

The effects of varying the humidity, pressure, voltage and time of applying the voltage, and the effect of electrolytes normally present on the transverse resistance of yarns and cloths were studied. A special humidity box which allows the samples to be conditioned and measured without opening is described. Data are given showing that the isoelec. point of silk as detd. by its resistance occurs near a pH of 4.2. (Copied complete from *Chem. Abs.*, 1935, V. 29, Col. 3523.) (W)

Electrometric Method of Determination of Amino Dyes. A. A. Morozov. *J. Gen. Chem.* (U. S. S. R.), 1934, V. 4, P. 1262–9.

Electrometric titration of *p*-phenylenediamine with HCl gave accurate quant. results. Titration curve shows a sharp break when 1 mol. of HCl was absorbed; the total HCl absorbed was 2 mols. per 1 mol. of the dye. (Copied complete from *Chem. Abs.*, 1935, V. 29, Col. 3520.) (W)

Rayon Yarns: Determination of the Changes in the Thickness of—Due to Stretching and Swelling. Karl Küsebach. *Mell. Textilber.*, 1934, V. 15, P. 472, 518–20, 562–5; *Chem. Abs.*, 1935, V. 29, Col. 3523.

The Rapid-Lanometer, a thickness gage, developed originally for wool fibres, is described. It can also be used for rayon. The Rapid-Lanometer measures the apparent cross-section of the yarn, including the air space between the fibres. An equation is given by which the true cross-section of the yarn can be calcd. (W)

Stains to Distinguish Fibroin and Silk Gum. W. S. Denham and E. Dickinson. *J. Soc. Dyers Col.*, 1935, V. 51, P. 93–7; *Chem. Abs.*, 1935, V. 29, Col. 3522.

The prepn. and staining of hanks and section of silk are outlined and observations reported on stains already in use, as well as in several other stains. Observations were made on the application of differentiating stains in the detection of residual gum on degummed silk. (W)

Waterproof Fabrics: Testing of. Francis D. Toyne. *Ind. Chemist*, 1934, V. 10, P. 380.

H_2O (60 0.1-g. drops/min.) falls through 6 ft. onto a 4-in. square of the cloth, which is stretched by four 20-g. weights over the mouth of a funnel; this delivers into a buret, and the time required to pass five ml. is recorded as the "proof-value." (Copied complete from *Chem. Abs.*, 1935, V. 29, Col. 3171.) (W)